Reactions of 2-Methylchromones with Cyanoacetamides and Ethyl Cyanoacetate. Synthesis of 6-(2-Hydroxyaryl)-4-methyl-2-oxo-1, 2-dihydropyridine-3-carbonitriles and 7-Hydroxy-6-imino-9-methyl-6*H*-benzo[*c*]chromene-8-carbonitriles

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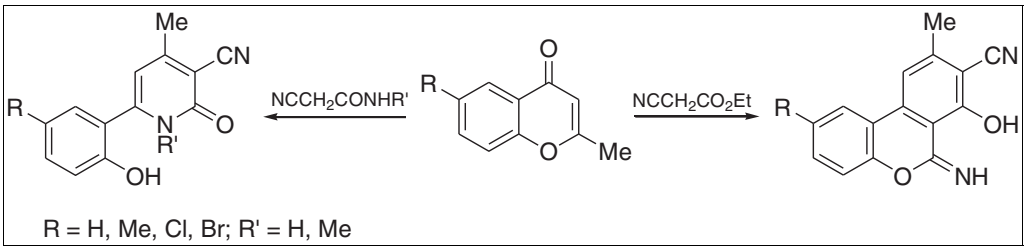

Although 2-methylchromones react with cyanoacetamide and *N*-methyl cyanoacetamide in the presence of sodium ethoxide in refluxing ethanol to produce 6-(2-hydroxyaryl)-4-methyl-2-oxo-1,2-dihydropyridine-3-carbonitriles, their reactions with ethyl cyanoacetate under the same conditions took an entirely different course and gave 7-hydroxy-6-imino-9-methyl-6*H*-benzo[*c*]chromene-8-carbonitriles.

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INTRODUCTION

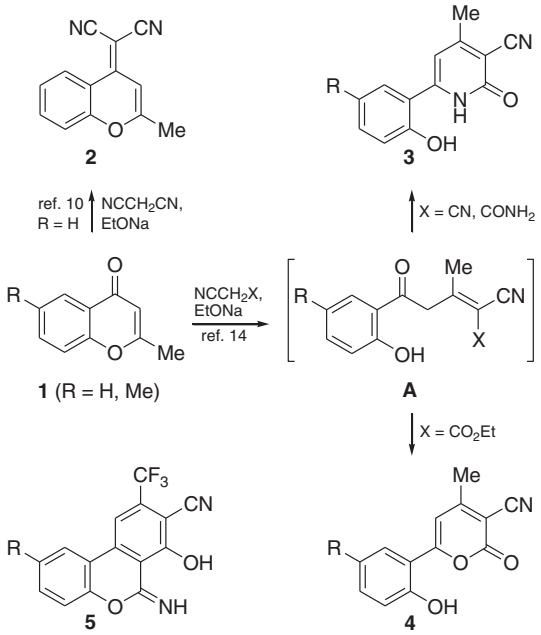
Chromones are naturally occurring oxygen-containing heterocyclic compounds that perform important biological functions in nature [1]. Although 2-methylchromones **1** constitute a small family of naturally occurring compounds, their synthesis and transformations into other biologically active compounds have been exploited [2,3]. It is known that the pyrone ring of **1** is susceptible to ring opening under the action of *N*-nucleophiles such as amines [4], hydrazines [5,6], hydroxylamine [6,7], and thiourea [8]. However, only a handful of papers describing some examples of reactions with *C*-nucleophiles is present in the literature. In almost all cases, the reactions with active methylene compounds proceeded without cleavage of the pyrone ring, and only 1,2-addition (Knoevenagel condensation) at the carbonyl group took place to give the corresponding methylidene derivatives [9,10], including compound **2** [10] (Scheme 1). In addition, 1,4-dianion generated from acetophenone oxime and phenyl magnesium bromide reacts with **1** at C-4 to give the spiroisoxazoline derivative [11] and 2-methyl-4-phenylchromen-4-ol [12], respectively.

On the other hand, there are only two reports on the reactions of 2-methylchromones **1** with *C*-nucleophiles leading to the products, which can result from the initial nucleophilic 1,4-addition. The use of methylcopper-BF_3 provided 2,2-dimethylchroman-4-ones via conjugate addition to the double bond in the γ-pyrone system [13]. Recently, the reaction of **1** with malononitrile, cyanoacetamide, and ethyl cyanoacetate in the presence of sodium ethoxide, leading to isolation of 2-pyridones **3** and 2-pyrones **4**, was reported [14]. Contrary to a previous report [10], the reaction proceeded via nucleophilic 1,4-addition with concomitant opening of the pyrone ring and subsequent intramolecular cyclization (Scheme 1). In view of these contradictory literature reports and our continued interest in the chemistry of chromones [15], we repeated the reaction of 2-methylchromones **1** with active methylene compounds following the literature method [10,14].

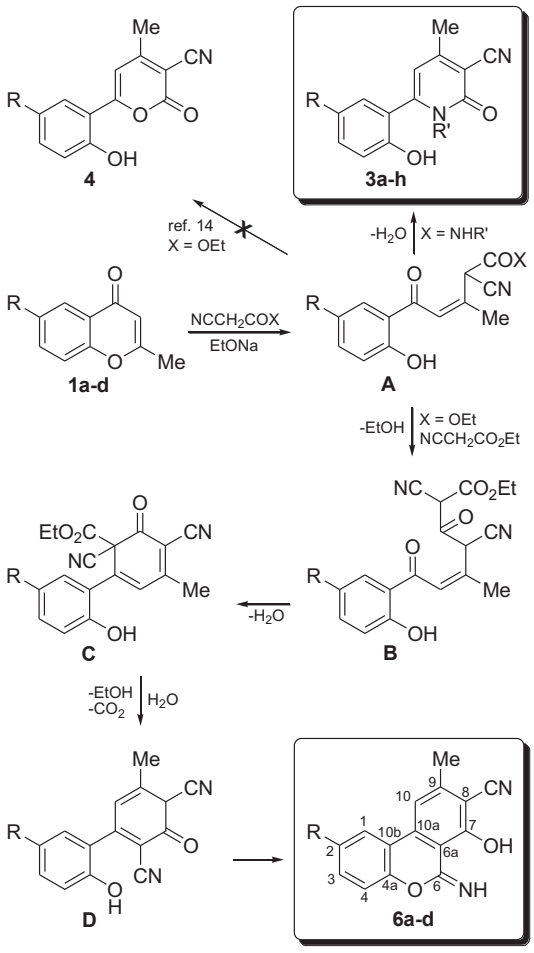
RESULTS AND DISCUSSION

We found that 2-methylchromones **1a–d** react with cyanoacetamide and *N*-methyl cyanoacetamide in the presence of sodium ethoxide in absolute ethanol (reflux, 10 h), affording 6-(2-hydroxyaryl)-4-methyl-2-oxo-1,2-dihydropyridine-3-carbonitriles **3a–h** in 47–76% yields. This reaction is earlier known transformation of 2-methylchromones **1a,b** [14], which should be regarded as nucleophilic 1,4-addition of cyanoacetamides with the formation of a new C–C bond (intermediate **A**) followed by cyclodehydration into the products **3** (Scheme 2). All the signals in the ^{1}H and ^{13}C NMR spectra of compound **3b** were assigned on the basis of 2D ^{1}H–^{13}C HSQC and HMBC experiments. Besides the signals expected for the aromatic protons, the ^{1}H NMR spectrum of **3b** in DMSO-d_6 showed four singlets because of the Me and MeN groups (δ 2.38 and 3.23), H-

Scheme 1



Scheme 2



3	R	R'	3	R	R'	1, 6	R
a	H	H	**e**	Cl	H	**a**	H
b	H	Me	**f**	Cl	Me	**b**	Me
c	Me	H	**g**	Br	H	**c**	Cl
d	Me	Me	**h**	Br	Me	**d**	Br

5 proton (δ 6.30), and phenolic hydroxyl (δ 10.27). In addition, the choice between 4-Me- and 6-Me-2-pyridones was made in favor of the former on the basis of the 2D HMBC spectrum for **3b**. The most informative cross-peaks are as follows: Me/C3, Me/C5, Me/CN, Me/C4. Note that in the case of 6-Me-2-pyridone, cross-peaks Me/C3 and Me/CN could not be observed.

As mentioned earlier, the previous workers [14] reported that the reaction of **1a,b** with ethyl cyanoacetate leads to compounds **4** (R═H, Me), and their claim has been favorably reviewed [2]. However, the 2-pyrone ring formation was not firmly established, and in light of the known behavior 2-(trifluoromethyl)chromones in reaction with ethyl cyanoacetate, which afforded 7-hydroxy-6-imino-9-(trifluoromethyl)-6*H*-benzo[*c*]chromene-8-carbonitriles **5** (Scheme 1) [16], it was anticipated that the process would follow a 1:2 instead of a 1:1 stoichiometry and proceed by double nucleophilic attack of the base-activated ethyl cyanoacetate to the chromone system. We therefore decided to repeat the condensation of chromones **1a–d** with ethyl cyanoacetate as originally reported [14].

It was found that the reaction of **1a** with ethyl cyanoacetate (2 equiv.) in the presence of sodium ethoxide in refluxing absolute ethanol gave a 61% yield of high melting solid with low solubility in solvents such as DMSO, DMF, ethanol, acetone, and toluene. This compound was described by Ibrahim *et al.* [14] as the α-pyrone **4** (R═H). However, in contrast with this structure, its composition corresponded to a 1:2 adduct, $C_{15}H_{10}N_2O_2$, which was assigned the structure 7-hydroxy-6-imino-9-methyl-6*H*-benzo[*c*]chromene-8-carbonitrile (**6a**) on the basis of elemental analysis, ^{1}H NMR, ^{13}C NMR, IR spectroscopies, and comparison of the spectroscopic data with the data reported for related systems [16]. In particular, the aromatic protons of compound **6a** at δ 7.37 (H-4), 7.40 (H-2), 7.61 (H-3), and 8.26 (H-1) compare well with those of trifluoromethylated analog, 7-hydroxy-6-imino-9-(trifluoromethyl)-6*H*-benzo[*c*]chromene-8-carbonitrile (**5**, R═H, δ 7.41, 7.43, 7.67, and 8.38, respectively). In addition, the Me group and H-10 appeared as two singlets at δ 2.53 and 7.52 ppm; the broad signal at δ 7.70 (2H) is due to the resonances of OH and NH protons. The Ibrahim group attributed the ^{1}H NMR aromatic signals at δ 7.37–8.29 ppm to the phenolic protons of **4** (R═H), although they are more befitting of a

coumarin system. Consequently, structure **4** should be revised to **6** (Scheme 2).

Similar reactions of **1b–d** with ethyl cyanoacetate gave 7-hydroxy-6-imino-9-methyl-6*H*-benzo[*c*]chromene-8-carbonitriles **6b–d** in 62–78% yields as the sole products after recrystallization from DMF. It should be noted that these 1:2 adducts were obtained even from the reaction of **1** with 1 equiv. of ethyl cyanoacetate, albeit in a lower yield. It was also found that formation of the tricyclic compounds **6** was encouraged by prolonged heating (12–16 h). Unfortunately, the low solubility of **6a–d** (mp >300°C) in suitable solvents made it difficult to obtain good ^{13}C NMR spectra; however, rather satisfactory ^{13}C NMR spectra obtained for compounds **6a**,**c** at 50°C supported the aforementioned structures. Additional support for **6** is provided by the IR spectra, in which a highly characteristic nitrile absorption at 2208–2216 cm^{-1} and C=NH and OH bands at 1678–1698, 3321–3326, and 3420–3428 cm^{-1} was observed.

Thus, the reaction of **1** with ethyl cyanoacetate took an entirely different course as compared with the reaction of cyanoacetamides. Clearly, the most significant difference between these reactions is the failure of cyanoacetamides to form 1:2 adducts. This failure may be attributed to the readiness with which 1:1 adducts undergo ring closure involving the amide group to form 2-pyridones **3**. In this process, ethyl cyanoacetate may be regarded as a synthetic equivalent of 1,3-dicyanoacetone, which is not a readily available compound. It should be noted that the products of type **2**, which can result from the initial nucleophilic 1,2-addition, were not observed at all.

For the formation of products **6**, a plausible mechanism depicted in Scheme 2 can be proposed. The initial nucleophilic attack of the base-activated methylene compound at C-2 (intermediate **A**) followed by Claisen condensation (intermediate **B**, the initial Claisen self-condensation of the starting ester could not be excluded), intramolecular cyclization and dehydration (intermediate **C**), and then aromatization (intermediate **D**, after hydrolysis and decarboxylation) leads to benzo[*c*]coumarins **6** through involvement of the phenolic hydroxy group.

In conclusion, we have shown that the condensation of 2-methylchromones with active methylene compounds in the presence of sodium ethoxide affords two types of products: 6-(2-hydroxyaryl)-3-cyano-4-methyl-2-pyridones with cyanoacetamides and 7-hydroxy-6-imino-9-methyl-6*H*-benzo[*c*]chromene-8-carbonitriles with ethyl cyanoacetate. The latter reaction represents a one-pot, multistep transformation and can be employed to obtain functionalized 6*H*-benzo[*c*]-chromen-6-ones.

EXPERIMENTAL

^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra were recorded on a Bruker Avance II spectrometer in DMSO-d_6 with TMS as an internal standard (only for ^{1}H NMR spectra). IR spectra were recorded on a Bruker Alpha FTIR instrument with the appliance of disturbed total internal reflection (ZnSe crystal). Elemental analyses were performed at the Microanalysis Services of the Institute of Organic Synthesis, Ural Branch, Russian Academy of Sciences. Melting points were uncorrected. All solvents used were dried and distilled per standard procedures.

General procedure for the synthesis of 6-(2-hydroxyaryl)-4-methyl-2-oxo-1,2-dihydropyridine-3-carbonitriles (3a–h). A solution of sodium ethoxide (69 mg, 3.0 mmol sodium in 5 mL of absolute ethanol) was added with the corresponding 2-methylchromone **1** (3.0 mmol) in absolute ethanol (5 mL) and cyanoacetamide or *N*-methyl cyanoacetamide (3.0 mmol). The mixture was refluxed for 10 h. Then, the orange reaction mixture was poured onto dilute hydrochloric acid (2N, 25 mL), and the yellow solid so formed was filtered and washed with ethanol to give compound **3** in an analytically pure state without recrystallization.

6-(2-Hydroxyphenyl)-4-methyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (3a). Yield 55%, light-brown crystals, mp >300°C (lit. [14] mp >300°C); IR (ATR) 3182, 2222, 1645, 1597, 1531 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) 2.40 (s, 3H, Me), 6.60 (br s, 1H, H-5), 6.92 (t, 1H, H-5′, J = 7.4 Hz), 6.98 (d, 1H, H-3′, J = 7.8 Hz), 7.34 (ddd, 1H, H-4′, J = 8.2, 7.0, 1.7 Hz), 7.46 (br s, 1H, H-6′), 10.55 (br s, 1H, OH), 12.15 (br s, 1H, NH).

6-(2-Hydroxyphenyl)-1,4-dimethyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (3b). Yield 56%, light-brown crystals, mp 260–263°C; IR (ATR) 3115, 2221, 1623, 1599, 1568, 1549 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) 2.38 (s, 3H, Me), 3.23 (s, 3H, MeN), 6.30 (s, 1H, H-5), 6.95 (td, 1H, H-5′, J = 7.4, 0.8 Hz), 6.99 (d, 1H, H-3′, J = 8.2 Hz), 7.23 (dd, 1H, H-6′, J = 7.6, 1.7 Hz), 7.38 (ddd, 1H, H-4′, J = 8.2, 7.2, 1.7 Hz), 10.27 (s, 1H, OH); ^{13}C NMR (100 MHz, DMSO-d_6) δ 20.8 (Me), 33.6 (Me), 100.7 (C-3), 110.7 (C-5), 116.2 (C-3′), 116.5 (CN), 119.9 (C-5′), 121.9 (C-1′), 130.1 (C-6′), 132.2 (C-4′), 152.9 (C-6), 154.8 (C-2′), 158.8 (C-4), 160.8 (C-2). *Anal*. Calcd for $C_{14}H_{12}N_2O_2$: C, 69.99; H, 5.03; N, 11.66. Found: C, 69.87; H, 4.91; N, 11.47.

6-(2-Hydroxy-5-methylphenyl)-4-methyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (3c). Yield 47%, light-brown crystals, mp >300°C (lit. [14] mp >300°C); IR (ATR) 3139, 2324, 2218, 1634, 1604, 1583 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) 2.24 (s, 3H, Me-5′), 2.40 (s, 3H, Me-4), 6.56 (br s, 1H, H-5), 6.87 (d, 1H, H-3′, J = 8.3 Hz), 7.15 (dd, 1H, H-4′, J = 8.3, 1.7 Hz), 7.28 (br s, 1H, H-6′), 10.27 (br s, 1H, OH), 12.10 (br s, 1H, NH). *Anal*. Calcd for $C_{14}H_{12}N_2O_2$: C, 69.99; H, 5.03; N, 11.66. Found: C, 70.02; H, 5.15; N, 11.76.

6-(2-Hydroxy-5-methylphenyl)-1,4-dimethyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (3d). Yield 63%, colorless crystals, mp 283−285°C; IR (ATR) 3162, 2220, 1624, 1612, 1597, 1567, 1549, 1512 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) 2.24, 2.38 (both s, 3H, Me), 3.23 (s, 3H, MeN), 6.30 (s, 1H, H-5), 6.88 (d, 1H, H-3′, J = 8.3 Hz), 7.04 (d, 1H, H-6′, J = 2.0 Hz), 7.18 (dd, 1H, H-4′, J = 8.3, 2.0 Hz), 10.02 (s, 1H, OH). *Anal*. Calcd for $C_{15}H_{14}N_2O_2$: C, 70.85; H, 5.55; N, 11.02. Found: C, 70.66; H, 5.78; N, 11.13.

6-(5-Chloro-2-hydroxyphenyl)-4-methyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (3e). Yield 69%, pale-yellow crystals, mp >330°C; IR (ATR) 3153, 3108, 2390, 2221, 1638, 1616, 1585, 1540 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) 2.44 (s, 3H, Me), 6.56 (br s, 1H, H-5), 6.95 (d, 1H, H-3′, J = 8.7 Hz), 7.25 (dd, 1H, H-4′, J = 8.7, 2.7 Hz), 7.48 (br s, 1H, H-6′), 10.63 (br s,

1H, OH), 12.10 (br s, 1H, NH). *Anal*. Calcd for $C_{13}H_9ClN_2O_2$: C, 59.90; H, 3.48; N, 10.75. Found: C, 59.74; H, 3.63; N, 10.97.

6-(5-Chloro-2-hydroxyphenyl)-1,4-dimethyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (3f). Yield 67%, light-brown crystals, mp 274−277°C; IR (ATR) 3078, 2580, 2230, 1601, 1561, 1493 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) 2.40 (s, 3H, Me), 3.28 (s, 3H, MeN), 6.21 (s, 1H, H-5), 6.96 (d, 1H, H-3′, J = 8.7 Hz), 7.19 (d, 1H, H-6′, J = 2.7 Hz), 7.30 (dd, 1H, H-4′, J = 8.7, 2.7 Hz), 10.44 (s, 1H, OH). *Anal*. Calcd for $C_{14}H_{11}ClN_2O_2$: C, 61.21; H, 4.04; N, 10.20. Found: C, 60.93; H, 4.33; N, 10.25.

6-(5-Bromo-2-hydroxyphenyl)-4-methyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (3g). Yield 76%, light-yellow crystals, mp >330°C; IR (ATR) 3155, 2500, 2220, 1640, 1616, 1591, 1539 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) 2.42 (s, 3H, Me), 6.52 (br s, 1H, H-5), 6.91 (d, 1H, H-3′, J = 8.7 Hz), 7.42 (dd, 1H, H-4′, J = 8.7, 2.5 Hz), 7.56 (br s, 1H, H-6′), 10.65 (br s, 1H, OH), 12.13 (br s, 1H, NH). *Anal*. Calcd for $C_{13}H_9BrN_2O_2 \cdot 0.25H_2O$: C, 50.43; H, 3.09; N, 9.05. Found: C, 50.23; H, 2.83; N, 8.91.

6-(5-Bromo-2-hydroxyphenyl)-1,4-dimethyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (3h). Yield 68%, brown crystals, mp 272−273°C; IR (ATR) 3138, 2221, 1624, 1590, 1571, 1547 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) 2.39 (s, 3H, Me), 3.26 (s, 3H, MeN), 6.24 (s, 1H, H-5), 6.92 (d, 1H, H-3′, J = 8.7 Hz), 7.34 (d, 1H, H-6′, J = 2.6 Hz), 7.45 (dd, 1H, H-4′, J = 8.7, 2.6 Hz), 10.50 (s, 1H, OH). *Anal*. Calcd for $C_{14}H_{11}BrN_2O_2 \cdot 0.25H_2O$: C, 51.96; H, 3.58; N, 8.66. Found: C, 51.69; H, 3.08; N, 8.53.

General procedure for the synthesis of 7-hydroxy-6-imino-9-methyl-6*H*-benzo[*c*]chromene-8-carbonitriles (6a–d). A solution of sodium ethoxide (69 mg, 3.0 mmol sodium in 5 mL of absolute ethanol) was added with the corresponding 2-methylchromone **1** (3.0 mmol) in absolute ethanol (5 mL) and ethyl cyanoacetate (680 mg, 6.0 mmol). The red-orange reaction mixture was refluxed for 12–16 h, cooled, and acidified with dilute hydrochloric acid (6N, 25 mL). The solid obtained was filtered off and crystallized from DMF to give compound **6**.

7-Hydroxy-6-imino-9-methyl-6H-benzo[c]chromene-8-carbonitrile (6a). Yield 61%, pale-yellow crystals, mp >300°C (lit. [14] mp >300°C); IR (ATR) 3428, 3326, 2211, 1698, 1599, 1581 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6, 50°C) δ 2.53 (s, 3H, Me), 7.37 (d, 1H, H-4, J = 8.3 Hz), 7.40 (t, 1H, H-2, J = 7.5 Hz), 7.52 (s, 1H, H-10), 7.61 (t, 1H, H-3, J = 7.8 Hz), 7.70 (br s, 2H, NH, OH), 8.26 (d, 1H, H-1, J = 7.5 Hz); ^{13}C NMR (100 MHz, DMSO-d_6, 50°C) δ 21.1 (Me), 95.7 (C), 101.1 (C), 110.3 (CH), 115.7 (C), 116.8 (CH), 116.9 (CN), 124.5 (CH), 124.7 (CH), 131.9 (CH), 139.2 (C), 150.0 (C), 151.1 (C), 153.7 (C), 160.9 (C). *Anal*. Calcd for $C_{15}H_{10}N_2O_2$: C, 71.99; H, 4.03; N, 11.19. Found: C, 71.68; H, 3.99; N, 11.32.

7-Hydroxy-6-imino-2,9-dimethyl-6H-benzo[c]chromene-8-carbonitrile (6b). Yield 62%, pale-yellow crystals, mp >300°C (lit. [14] mp >300°C); IR (ATR) 3420, 3322, 2208, 1678, 1583 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 2.44 (s, 3H, Me-2), 2.55 (s, 3H, Me-9), 7.22 (d, 1H, H-4, J = 8.3 Hz), 7.38 (br d, 1H, H-3, J = 8.3 Hz), 7.44 (s, 1H, H-10), 7.6–8.0 (br s, 1.6H, NH, OH), 8.01 (br s, 1H, H-1), 8.19 (s, 0.4H, NH). *Anal*. Calcd for $C_{16}H_{12}N_2O_2$: C, 72.72; H, 4.58; N, 10.60. Found: C, 72.78; H, 4.54; N, 10.64.

2-Chloro-7-hydroxy-6-imino-9-methyl-6H-benzo[c]chromene-8-carbonitrile (6c). Yield 78%, pale-yellow crystals, mp >300°C; IR (KBr) 3422, 3321, 2216, 1687, 1612, 1598, 1574 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6, 50°C) δ 2.55 (s, 3H, Me), 7.37 (d, 1H, H-4, J = 8.9 Hz), 7.53 (s, 1H, H-10), 7.55 (dd, 1H, H-3, J = 8.9, 2.4 Hz), 7.6–7.9 (br s, 1H, OH), 8.17 (s, 1H, NH), 8.29 (d, 1H, H-1, J = 2.4 Hz); ^{13}C NMR (100 MHz, DMSO-d_6, 50°C) δ 21.7 (Me), 95.9 (C), 111.4 (CH), 116.3 (CN), 119.4 (C), 119.6 (CH), 124.7 (CH), 129.7 (C), 132.1 (C), 132.2 (CH), 138.7 (C), 151.0 (C), 152.6 (C), 154.3 (C), 161.3 (C). *Anal*. Calcd for $C_{15}H_9ClN_2O_2$: C, 63.28; H, 3.19; N, 9.84. Found: C, 63.26; H, 3.19; N, 9.82.

2-Bromo-7-hydroxy-6-imino-9-methyl-6H-benzo[c]chromene-8-carbonitrile (6d). Yield 71%, pale-yellow crystals, mp >300°C; IR (KBr) 3424, 3324, 2213, 1687, 1611, 1595, 1575 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 2.55 (s, 3H, Me), 7.31 (d, 1H, H-4, J = 8.8 Hz), 7.54 (s, 1H, H-10), 7.68 (dd, 1H, H-3, J = 8.8, 2.3 Hz), 7.6–7.9 (br s, 1.5H, OH), 8.17 (s, 0.5H, NH), 8.41 (d, 1H, H-1, J = 2.3 Hz). *Anal*. Calcd for $C_{15}H_9BrN_2O_2$: C, 54.74; H, 2.76; N, 8.51. Found: C, 54.46; H, 2.69; N, 8.44.

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